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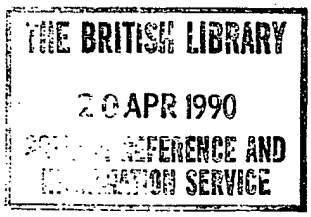
COMPLETE SPECIFICATION

(54) PROCESS FOR PREPARING LONG-PRESERVATION AROMATIZED DRINKS BASED ON ACID WHEY



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The present invention relates to a novel process for preparing aromatized beverages for long preservation, based on acidified milk whey herein refered to as acid whey.

Conventional processes for manufacturing long-preservation aromatized beverages, based on acid whey employ:

- on the one hand, acidification of the whey to lower the pH thereof to a value equal to 4.6 or slightly below the latter, either by using lactic ferments, or by the addition of edible acids :

- on the other hand, sterilizing heat treatment at high temperature, generally from 120 to 140°C, necessary for the complete destruction of the microbial flora which are developed in acid whey, in order to ensure long preservation of the latter.

However, the heat treatment of acid whey in the pH zone indicated presents the drawback of causing the precipitation of the soluble proteins contained in acid whey at temperatures above or equal to 100°C.

To overcome this drawback, it has already been proposed to incorporate stabilizers compatible with the acid pH, such as alginates, carboxy methylcellulose, guar gum, etc., in the acid whey and to apply to the latter a homogenization treatment prior to its heat treatment.

However, the use of a stabilizer introduces a non-dairy substance into the whey, and the homogenization treatment constitutes an expensive technology.

Now, Applicants have observed that it is possible to render the acid whey heat stable at high temperatures which can reach 160°C by lowering its pH to a value equal to or less than 3.8.

However, it is very difficult to achieve pH values equal to or less than 3.8 by lactic fermentation ; in addition, the latter method necessitates the storage of large amounts of raw material for long periods and

the management of the fermentation is always delicate.

On the other hand, it is easy to acidify the whey at pH equal to or less than 3.8 by the addition of an edible acid; however, this technology necessitates the use of a relatively large amount of acid. Thus, 1 litre of whey from cheese-making of which the initial pH is 6.2 requires the addition of more than 5g of citric acid to reach a pH of 3.6.

Moreover, the original inorganic substances in the whey exert a buffer effect with respect to the edible acid added, so that it is necessary to add large amounts of acid to reach the low pH desired and the final aromatized product has a pronounced disagreeable acid and salty taste.

It is therefore an object of the present invention to provide a process for preparing long-preservation aromatized beverages, based on heat stable acid whey, without the addition of a stabilizer and having a pleasant flavour, without an acid and salty taste.

It is also an object of the invention to provide a process for performing in a single operation:

- the acidification of the whey at a pH equal to or less than 3.8 to confer on said product good heat stability,
- and sufficient decationization to eliminate therefrom the unpleasant acid and salty tastes, and to permit a wider choice of flavours.

The process according to the invention comprises a step of simultaneous acidification and decationization of the whey by treating said whey with a cation exchange resin in acid form, or by electrodialysis on cationic membranes, for a time necessary to lower the pH of said whey to a value equal to or less than 3.8, preferably from 3.2 - 3.8, followed by a heat treatment step of the acidified and decationized product obtained, at a temperature of 100 to 160°C, and more particularly at 120-140°C to sterilize it.

If necessary, sweetening agents and flavouring substances are added to the acidified and decationised whey prior to the heat treatment of the latter.

As whey, any type of milk whey may be used and preferably mild wheys from cheese-making derived from the manufacture of pressed cheeses, cooked cheeses or soft cheeses, and casein wheys obtained by the exchange process.

The whey can be enriched in proteins by using known technologies such as ultrafiltration or the addition of soluble whey proteins.

The whey can also be partly delactosed by crystillisation of the lactose in order to provide a beverage less rich in sugar.

In the same way, the lactose of the whey may be hydrolysed into glucose and into galactose in order to provide a beverage acceptable by alactasic persons.

As cation exchange resins, it is possible to use cation exchange resins in the acid form, of conventional type, used in conventional demineralisation.

In particular, it is possible to use according to the invention highly acid cation exchange resins with a polystyrene-divinyl benzene skeleton bearing sulfonic acid groups and having the form of gels or macroporous solids (beads,rods,etc.).

Highly acid cationic resins are marketed under the following trademarks:

"Duolite" of Diaprosim-Diamond;
-"Amberlite" of Rohm and Haas;
-"Lewatit" of Bayer
-"Kastel" of Montedison;
-"Dowex" of Dow Chemical;
-"Relite" of Residion;

The following highly cationic resins are preferred:

- "Lewatit S 100 and SP 112";
- "Duolite C 20 and C 26";
- "Amberlite IR 120 and 200";
- "Kastel C 300 and C 300 P";
- "Dowex HCR-S and MSC-1".

It is also possible to use weakly acid cationic resins, with carboxylic acid functional groups.

The preferred weakly cationic resins are the following:

- "Kastel C10";
- "Duolite C 464";
- "Amberlite IRC 84";
- "Lewatit CNP";
- "Dowex CCR-2".

The treatment of the whey with the cation exchange resin can be carried out by techniques known in themselves, for example, by percolation through a fixed bed of exchange resin or by mechanical or pneumatic stirring of the whey - cationic exchange resin mixture.

After saturation of the cationic exchange resin with the whey cations, a regeneration of said exchange resin by an acid follows. After rinsing the exchange resin thus regenerated with decationised water, the regenerated exchange resin is ready for a further processing cycle.

The preferential interval of pH acidification of the whey is from 3.2 to 3.8.

For a pH above 3.8, the whey is not heat stable at temperatures above or equal to 100°C.

For a pH less than 3.2, the whey takes an unpleasant acid taste.

The range of the treatment temperatures of the whey is from 100 to 160°C; at these temperatures, the totality of the microbial germs contained in the whey is destroyed without the soluble proteins being precipitated whilst pasteurisation at 72°C only kills a part of these germs.

The general operational method for the practising of the process according to the present invention is described below.

The mild whey, derived from cheese manufacturers, is freed from soft curds by centrifugal separation. After cooling, to a temperature below 10°C, to avoid bacterial growth, the whey is subjected to cation exchange with a cationic resin in the H^+ form.

It is possible to use a highly acid cationic acid with sulfonic groups or a weakly acid cationic resin with carboxylic groups.

In using a highly acid cationic resin, in H^+ form, the whey is acifified to a pH below 2.0, then mixed in suitable proportions, with unacidified whey, so that the mixture arrives at a pH comprised between 3.2 and 3.8, preferably between 3.4 and 3.6. The proportions are generally comprised between 1 and 1.6 parts of unacidified whey per part of acidified whey.

In the use of a weakly acid cationic resin, in the H^+ form, the whey is acidified until the average pH reaches the desired value (pH 3.2-3.8, preferably 3.4-3.6), at which time the processing cycle is stopped.

The acidified whey so obtained is supplemented with a sufficient amount of sugar and flavouring substances. The sweetened and flavoured mixture is subjected to U.H.T sterilization between 100 and 160°C, preferably between 120 and 140°C, and packaged aseptically. (U.H.T. = ultra high temperature).

According to a modification of the process of the invention, one part of the whey is subjected to ultrafiltration treatment to the desired protein concentration level then acidified by mixing with previously acidified and decationised whey in the proportions defined by the desired protein concentration and by the value of the pH desired for the final mixture.

According to another modification of the process, the whey is concentrated to bring the lactose that it contains to a super-saturation level such that the lactose can crystallize. The crystallised lactose is separated

from the whey. The delactosed whey is diluted in a sufficient amount of water (substantially equivalent to the water evaporated during the concentration operation), so that its content of inorganic salts comes back to the same value as that in the initial whey. The product is acidified as described previously then aromatised and subjected to the sterilization treatment.

The following non-limiting examples are given to illustrate the manufacture of aromatised beverages based on acidified whey, containing proteins, without the addition of stabilizers, and for long preservation, according to the invention.

EXAMPLE 1

In this example, the preparation of an aromatised beverage from acidified whey by a weakly acid cation exchange resin, is described.

1,000 litres of whey, of pH 6.35, previously freed from soft curds by centrifugal separation and cooled to 10°C are treated by percolation through a bed of 40 litres of weakly acid cation exchange resin marketed under the name Dowex CCR-2 (with carboxylic acid groups), presented in the form of beads of 0.3 to 1.2 mm diametre. A decationised whey acidified to pH 3.55 is recovered ; the exhausted resin is washed with water and regenerated by a 6% hydrochloric acid solution.

75 kg of saccharose and 0.8 kg of apple flavouring are added to the acidified whey and the mixture so obtained is preheated in a plate exchanger to 85°C then sterilized indirectly at 135°C for 2 to 5 seconds. After cooling the product obtained is packaged aseptically.

The composition of the product analysed before its aromatisation and the results of physical and organoleptic examinations carried out on the product 48h after its packaging are collected in Table I below.

Table I

	Starting whey	Whey acidified by cation exchange
Dry matter g|l	64.0	62.4
Acidity °D (degree Domic)	14	40
pH	6.35	3.55
Calcium %DE (% of the dry extract)	0.69	0.06
Sodium %DE	0.65	0.65
Potassium % DE	2.6	2.25
Chloride %DE	2.90	2.95
Proteins g|l	8.9	8.75
Stability	-	good, no precipitation, no deposit
Taste	-	sweet, pleasant

EXAMPLE 2

In this example, the preparation of an aromatised beverage from whey acidified by a strongly acid cation exchange resin, is described.

450 litres of milk whey, of pH 6.3, freed from soft curds and cooled to 8°C are treated by percolation through a bed of 30 litres of strongly acid cation exchange resin of the gel type, marketed under the name Duolite C 20 (with sulfonic acid functionel groups).

A decationised whey acidified to pH 1.95 is recovered.

The latter is mixed with 550 litres of untreated, cold cheesemaking whey at 8°C, to obtain 1000 litres of whey acidified to pH 3.55.

After the addition of 75kg of saccharose and 800g of apple flavour, pre-heating to 85°C and sterilisation at 135° C the product obtained is packaged aseptically.

The specimens were examined and tasted 48h after packaging.

The composition of the analysed product before its aromatisation and the results of the physical and organoleptic examinations carried out on the product 48h after its packaging are grouped in Table II below.

Table II

	Starting Whey	Whey acidified by cation exchange
Dry matter g/l	64.6	62.5
pH	6.3	3.55
Acidity °D	14	41
Calcium % DE	0.68	0.45
Sodium %DE	0.75	0.50
Potassium %DE	2.5	1.75
Chlorides %DE (NaCl)	2.95	3.05
Proteins g/l (N X 6.38)	9.1	8.65
Appearance, colour	-	opalescent
Stability	-	good, no precip-. itation, no deposit
Taste	-	sweet, pleasant

EXAMPLE 3

In this example, the preparation of an aromatised beverage from whey acidificd by acid metathesis (or electrodialysis on cationic membranes) is described.

1,000 litres of milk whey acidified by cation exchange to pH 1.75 were mixed with 1250 litres of untreated cheese-making whey to obtain 2250 litres of whey acidified to pH 3.5. This mixture is subjected to ultrafiltration at 52°C. 1 000 litres of whey retentate were collected and subjected to aromatization and sterilization under the preceding conditions described in Example 1.

The composition of the product analyzed before its aromatization is given in Table IV below.

TABLE IV

	Starting whey	Whey acidified before ultrafiltration	Whey acidified and concentrated by ultra filtration
Dry material g|l	64.2	62.4	72.0
pH	6.3	3.5	3.47
Acidity °D	14	46	50
Calcium % DE	0.7	0.44	0.42 (300 mg|1)
Sodium % DE	0.75	0.49	0.39 (280 mg|1)
Potassium %DE	2.45	1.75	1.25 (900mg|1)
Chlorides %DE (NaCl)	2.90	2.95	2.75
Proteins g|l	9. 0	8.65	15.8 (22% of the dry matter)

The beverage enriched with whey proteins remains stable to preservation (6 months) and has a pleasant slightly milky taste.

In addition the acidity of the whey before ultrafiltration exerts a certain bacteriostatic activity during the whole ultrafiltration operation.

Comparative test A

By way of comparison, a whey acidified by lactic fermentation and stabilised by the addition of stabiliser was prepared.

To 1000 litres of milk whey freed from soft curd and cooled to 10°C, were added, with very vigorous stirring, 5kg of sodium alginate as stabiliser compatible with the low pH. The homogeneous mixture was pasteurised at 72 °C and immediately cooled on a plate exchanger to the incubation temperature. The fermentation was ensured by 20kg of a mixture of lactic ferments for 12h at 45° C to obtain pH of 4.2.

The stabilised acidified whey was cooled to 8 °C and 75kg of sugar and 800g of apple flavouring were added.

The mixture pre-heated to 85 ° C was homogenized under a pressure of 250 kg|cm2 then sterilised and packaged as described above in Example 1.

The composition of the product analysed before its aromatization and the results of the physical and organoleptic examinations carried out on the product 48 h after its packaging are grouped in Table V which follows.

TABLE V

	Starting whey (identical to that of Example 1)	Whey acidified by lactic fermentation and stabilised
Dry material g/l	64.0	63.0
Acidity °D	14	53
pH	6.35	4.2
Calcium %DE	0.69	0.68
Sodium %DE	0.65	0.65
Potassium %DE	2.6	2.6
Chlorides %DE	2.90	2.90
Proteins g/l	8.9	8.95
Stability	-	average (slight deposit)
Taste	-	acidulated

The drink obtained had an acceptable stability due to the addition of a stabiliser and to the homogenisation treatment. Content of titratable acid expressed in Dornic acidity was considerable and gave the product an acidulated taste which is further reinforced by the inorganic source present in considerable amount in the whey.

The beverage obtained by cation exchange in Example 1 had better taste properties and a stability at least equivalent to that of the product acidified by fermentation and stabilised by the addition of a stabiliser and homogenization without its manufacture involving these two operations.

When the comparative test described above is repeated, but without the addition of the stabiliser or homogenization the beverage acidified by fermentation immediately allows the soluble proteins to precipitate

TABLE V

	Starting whey (identical to that of Example 1)	Whey acidified by lactic fermentation and stabilised
Dry material g|l	64.0	63.0
Acidity °D	14	53
pH	6.35	4.2
Calcium %DE	0.69	0.68
Sodium %DE	0.65	0.65
Potassium %DE	2.6	2.6
Chlorides %DE	2.90	2.90
Proteins g|l	8.9	8.95
Stability	-	average (slight deposit)
Taste	-	acidulated

The drink obtained had an acceptable stability due to the addition of a stabiliser and to the homogenisation treatment. Content of titratable acid expressed in Dornic acidity was considerable and gave the product an acidulated taste which is further reinforced by the inorganic source present in considerable amount in the whey.

The beverage obtained by cation exchange in Example 1 had better taste properties and a stability at least equivalent to that of the product acidified by fermentation and stabilised by the addition of a stabiliser and homogenization without its manufacture involving these two operations.

When the comparative test described above is repeated, but without the addition of the stabiliser or homogenization the beverage acidified by fermentation immediately allows the soluble proteins to precipitate

on sterilisation at 135°C.

Comparative Test B

By way of comparison, there were also prepared :

- a whey acidified by lactic fermentation,
- and a whey acidified artificially by citric acid

1a) Natural acidification

1000 litres of milk whey were pasteurised at low temperature (63°C for 30 min) to avoid denaturation of the proteins then cooled to 45° C.

30 kg of mixture of lactic ferments (Streptococcus thermophilus + lactobacillus bulgaricus + lactobacillus helveticus) were added to the whey. After 16h of incubation, the pH reach the value of 3.7. The mixture was cooled to 8°C.

1b) Artificial Acidification

To 1,000 litres of milk whey cooled to 8°C were added 5.9 kg of citric acid, to reach the pH 3.6.

75 kg of saccharose and 800 g of apple flavouring were added to various acidified wheys.

After preheating in a plate exchanger to 85°C, the wheys were sterilised indirectly at 135 °C then packaged aseptically after cooling. The samples were examined and tested 48 h after packaging.

The composition of the products analysed before their aromatization and the results of the physical examinations carried out on the products 48 h after their packaging are also grouped on Table VI as follows.

TABLE VI

	Starting whey (identical to Example 2)	Whey acidified by lactic fermentation	Whey acidified by the addition of citric acid
Dry matter g/l	64.6	62	66.0
pH	6.3	3.7	3.60
Acidity °D	14	83	95
Calcium % DE	0.68	0.67	0.65
Sodium % DE	0.75	0.75	0.70
Potassium % DE	2.5	2.45	2.35
Chlorides % DE (NaCl)	2.95	2.90	2.75
Proteins g/£ (N X 6.38)	9.1	9.0	8.5
Stability		average	good
Taste		acid	pronounced acid

The beverages obtained in these tests had an average to good stability due to the fact that the pH of the whey had been lowered to 3.7 and to 3.6, but their measured acidity expressed in °Dornic was very strong (83°D for the product acidified by fermentation and 95°D for the product acidified by the addition of acid against 41°D only for the product obtained according to the invention in the Example 2) and, for this reason, these beverages had a disagreeably pronounced acid taste.

On the other hand, despite its acid pH (3.55), the whey acidified by cation exchange according to the invention in Example 2 does not confer on the aromatised beverage an acid taste, contrary to the products obtained by lactic fermentation or by the addition of citric acid ; taking into account the taste neutrality of the product obtained according to the invention, it is possible to aromatize it with an extended choice of flavours.

When the comparative tests described above are repeated, but acidifying the wheys to a pH higher than 3.8, for example, to pH 4.2, the acidified products allow the precipitation of the soluble proteins on sterilisation at 135°C.

CLAIMS

1. A process for the preparation of a long preservation aromatized beverage, based on acidified milk wheys, said process comprising the following steps :

- a step of simultaneous acidification and decationisation of the whey by treating said whey with a cation exchange resin in acid form, or by electrodialysis on cationic membranes, for a time necessary to lower the pH of said whey to a value equal to or less than 3.8 and to decationize at least partly,
- followed by a heat treatment step of the acidified and decationized product at a temperature of 100 to 160°C to sterilize the latter.

2. A process according to claim 1, wherein there are added to the whey acidified to a pH equal to or lessthan 3.8, sweetening and flavouring agents before its heat treatment.

3. A process according to claim 1 or 2, wherein the whey is acidified to a pH of 3.2 to 3.8.

4. A process according to claim 1 or 2, wherein the heat treatment is done at a temperature of 120 to 140°C.

5. A process according to claim 1, wherein the starting whey is at least partially of whey enriched in proteins by ultra-filtration or by the addition of soluble whey proteins.

6. A process according to claim 1, wherein the starting whey is at least partially delactosed whey.

7. A process according to claim 1, wherein the lactose contained in the starting whey has been previously hydrolyzed.

8. A process according to claim 1, wherein the cation exchange resin in acid form is a strongly acid resin bearing sulfonic acid groups or weakly acid resin bearing carboxylic acid groups.

9. A process according to one of claims 1,5,6 and 7, wherein the whey is acidified by cation exchange to a pH less than 2 and then mixed with unacidified whey in the proportions suitable to result in a pH of the mixture between 3.2 and 3.8.

10. A process according to claim 1 for the preparation of a long preservation aromatized beverage, substantially as hereinbefore described with particular reference to the accompanying Examples.

11. A long preservation aromatized beverage, based on acidified whey, obtained according to any one of the preceding claims 1 to 10.

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